DEFENSE TECHNICAL INFORMATION CENTER

Information for the Defense Community

DTIC® has determined on 7/27/2011 that this Technical Document has the Distribution Statement checked below. The current distribution for this document can be found in the DTIC® Technical Report Database.

☒ **DISTRIBUTION STATEMENT A.** Approved for public release; distribution is unlimited.

☐ **© COPYRIGHTED.** U.S. Government or Federal Rights License. All other rights and uses except those permitted by copyright law are reserved by the copyright owner.

☐ **DISTRIBUTION STATEMENT B.** Distribution authorized to U.S. Government agencies only (fill in reason) (date of determination). Other requests for this document shall be referred to (insert controlling DoD office).

☐ **DISTRIBUTION STATEMENT C.** Distribution authorized to U.S. Government Agencies and their contractors (fill in reason) (date determination). Other requests for this document shall be referred to (insert controlling DoD office).

☐ **DISTRIBUTION STATEMENT D.** Distribution authorized to the Department of Defense and U.S. DoD contractors only (fill in reason) (date of determination). Other requests shall be referred to (insert controlling DoD office).

☐ **DISTRIBUTION STATEMENT E.** Distribution authorized to DoD Components only (fill in reason) (date of determination). Other requests shall be referred to (insert controlling DoD office).

☐ **DISTRIBUTION STATEMENT F.** Further dissemination only as directed by (insert controlling DoD office) (date of determination) or higher DoD authority.

Distribution Statement F is also used when a document does not contain a distribution statement and no distribution statement can be determined.

☐ **DISTRIBUTION STATEMENT X.** Distribution authorized to U.S. Government Agencies and private individuals or enterprises eligible to obtain export-controlled technical data in accordance with DoDD 5230.25; (date of determination). DoD Controlling Office is (insert controlling DoD office).

Yttria Nano-Particle Reinforced CP Titanium

Cooperative Agreement between

FMW Composite Systems, Inc. AND **US Army Research Laboratory**

Agreement No.: W911NF-11-2-0003

CLIN 0001: **FIRST ANNUAL PROGRAM PLANNING FINAL REPORT**

Submitted by:

FMW Composite Systems, Inc.

Sesh Tamirisa, Program Manager

1200 W. Benedum Industrial Drive

Bridgeport, WV 26330

Tel: 304-842-1970

e-mail: stamirisa@fmwcomposite.com

To The ARL Cooperative Agreement Manager (CAM):

Vincent Hammond

U.S. Army Research Laboratory (ARL)

Building 4600, Room N102

Aberdeen Proving Ground, MD 21005

Tel: 410-306-0855

e-mail: vincent.h.hammond.civ@mail.mil

8 JULY 2011

SUMMARY

Literature shows that doubling of tensile properties is achievable via nano-sized yttria (Y_2O_3) dispersion in titanium. These initial studies reported proof of concept using small laboratory-scale samples produced via arc-melting but further studies to establish the technology and applications are lacking. The objective of this work is to study the scale-up feasibility of yttria dispersed ti tanium and to investigate the influence of yttria nano p articles on the tensile properties of titanium. The focus of this effort is to perform initial experiments to determine if this effect occurs in metal powder produced through gas atomization. In addition, a higher strength version of the material was sought through the addition of boron. This project is designed to explore the efficiency of nano-sized yttria at improving tensile properties of CP titanium for US Army applications. This phase of the project is focused on evaluating the tensile properties of a limited number of samples with specified compositions.

Commercial purity (CP) titanium and inert gas atomization were selected as material and process in this study. Scale-up feasibility of nano-yttria dispersed CP Ti was successfully demonstrated by producing large quantities of CP Ti alloys via conventional titanium powder metallurgy route. Three alloys, CP Ti, CP Ti + 0.3% Y_2O_3, and CP Ti + 0.3% Y_2O_3 + 0.5 B, were made. Powder compacts were fabricated via hot isostatic pressing and billets were extruded to produce 0.5" (12.7 mm) diameter bars. Room temperature tensile testing was performed on multiple specimens machined from bars. Addition of 0.3% Y_2O_3 increased the tensile strength of CP Ti by 25%. Addition of 0.5% B, in addition to 0.3% Y_2O_3, increased the tensile strength of CP Ti further up to 67%. Addition of 0.5% B also increased the tensile modulus of CP Ti by 20% in addition to the significant strength increase while maintaining adequate ductility. Yttria addition, on the other hand, provided only a strength benefit.

The feasibility of producing yttria dispersed titanium and improvements in tensile properties were successfully demonstrated. These alloy systems offer potential for significant weight reduction benefit for the US Army applications by taking advantage of improved strength and stiffness of CP Ti.

1. Fabrication of Yttria Nano-particle Reinforced CP Titanium

Pre-alloyed powders of commercial purity (CP) Ti, CP Ti + 0.3% yttria, and CP Ti + 0.3% yttria + 0.5% boron were produced via inert gas atomization technique. As-atomized powders were sieved to obtain −35 mesh (500 μm mesh opening size) fractions. Chemical analysis (wt.%) and sieve analysis of all three powder compositions are given in Figure 1. In comparison to typical CP Ti Grade 2 chemistry (0.3% max Fe and 0.25% max O), the Fe and O contents were lower in the powder materials produced in this study. However, these levels were similar in the baseline CP Ti and yttria modified CP Ti, which allows accurate comparison of properties.

ATI Powder Metals

6515 Steubenville Pike
Pittsburgh, PA 15205-1005
(412) 923-2955; FAX (412) 788-4665

Page 1 of 1

Date: February 25, 2011

CERTIFICATE OF TEST

CR Order: T802

Customer:	Purchase Order No:	Purchase Order Date:
FMW Composite Systems	11462	December 7, 2010

Specification:

Item No.:	Weight:	Product Description:
1	15.0 lbs	CP Ti Grade 2 Powder, -35 Mesh, Heat 4255
2	65.0 lbs	CP Ti Grade 2 + 0.45Y_2O_3 Powder, -35 Mesh, Blend B630
3	65.0 lbs	CP Ti Grade 2 + 0.45Y_2O_3 ,+ 0.5B Powder, -35 Mesh, Blend B631

Chemical Analysis (Wt%):

Lot	Fe	Y	B	C	O	N	H	Ti
4255	0.038	0.005	----	0.051	0.088	0.006	0.003	Bal.
B630	0.044	0.30	---	0.035	0.107	0.003	0.003	Bal.
B631	0.053	0.32	0.53	0.041	0.101	0.005	0.004	Bal.

Powder Sieve Analysis (% Under):

Lot	-35	-45	-60	-80	-100	-140	-200	-230	-270	-325	Tap Density (g/cc)	Flow Rate (sec)
4255	100.0	91.1	72.4	51.6	41.0	19.1	7.1	3.8	2.4	1.5	2.53	38
B630	100.0	88.4	65.5	46.0	38.2	21.4	11.2	7.2	5.0	3.3	2.91	33
B631	100.0	87.9	65.9	46.3	37.9	20.2	10.3	6.3	4.6	3.0	2.84	31

Note: The recording of false, fictitious, or fraudulent statements or entries on this document may be punishable as a felony under Federal Statutes. This test report shall not be reproduced except in full without the written approval of ATI Powder Metals.

Certified by:

Joseph F. Muha

Joseph F. Muha

2/25/11

Date

CR Form 4-003 Rev. 5 10/26/09

Figure 1: Chemical analysis and sieve analysis of yttria nano-particle reinforced CP Titanium powders.

2. Tensile Properties of Yttria Nano-particle Reinforced CP Titanium

Powders of about 10 lb from each composition were packed in mild steel cans, hot offgassed at 500°F for 2 hours, and vacuum sealed. Sealed cans were subjected to hot isostatic pressing (HIP) at 1750°F and 15 ksi for 3 h and fully dense powder compacts were produced. Compacts were decanned and machined into billets of 2.95" diameter and 7" height. Billets were extruded into 0.75" diameter bars using conditions of 1650°F billet temperature with 1 hour soak time, 12:1 extrusion ratio, 100 inch/min ram speed, and air cooling after extrusion. Pictures of powder cans, machined billets, and extruded bars are shown in Figure 2.

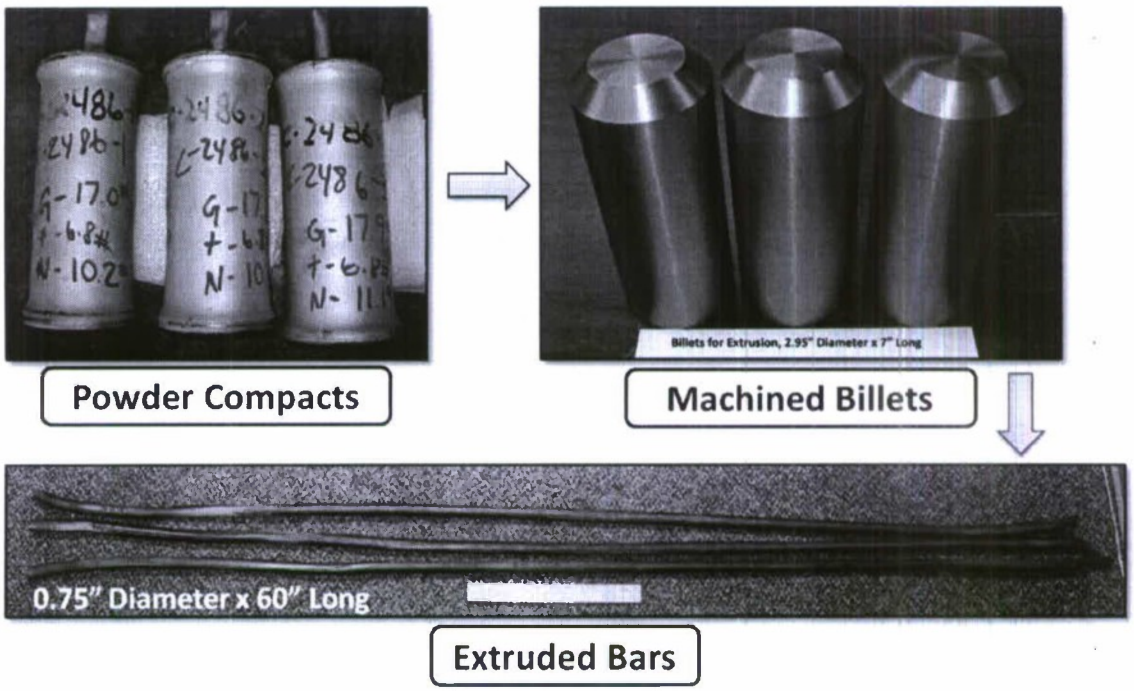

Figure 2: Fabrication process flow of yttria nano-particle reinforced CP Ti extruded bars.

Blanks of 3" length were cut from extrusions and tensile specimens of 0.25" gauge diameter and 1" gauge length were machined. Six samples from each alloy were machined to tensile specimen drawing shown in Figure 3. Tensile testing of specimens was performed at room temperature per the ASTM standard E8 using an initial ram speed of 0.005 in/in/min (0.00212 mm/s). Digital elongation data was recorded using an extensometer of 1" length attached to the gauge portion of the specimen.

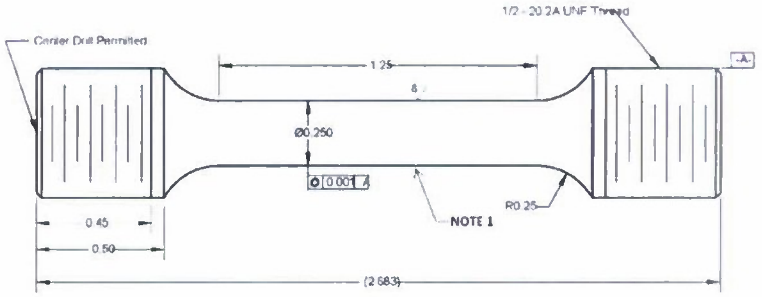

Figure 3: Tensile specimen drawing used for evaluating CP Ti extruded bars.

Tensile data generated on all three alloys are presented in Table 1. Tensile stress – strain curves of all tested samples are presented in Figures 4 – 6 (data on CP Ti and CP Ti + 0.3 Y_2O_3 samples were truncated as the number of data points exceeded the Excel plotting limit). Tensile strengths (0.2% yield and ultimate) of three alloys are compared in Figure 7. Addition of 0.3Y_2O_3 increased the tensile strength of CP Ti by 25% (+50 MPa in TYS and +80 MPa in UTS) with the tensile elongation value was unchanged. It was noted that tensile strengths were significantly lower and tensile elongations were higher compared to typical CP Ti Grade 2 (275 MPa guaranteed minimum tensile yield strength and 20% tensile elongation). These results are consistent with the lower Fe and O contents in powder materials. Addition of 0.5B increased the tensile strength further (52 – 67%, +131 MPa in TYS and +170 MPa in UTS) compared to the baseline CP Ti. Addition of B also increased the tensile modulus by 20% (+21 GPa) compared to the baseline CP Ti and yttria modified CP Ti as shown in Figure 8. In the B-modified CP Ti, a reduction in total tensile elongation to 28% occurred compared to the baseline alloy (Figure 9). However, the tensile elongation is well above the desired value of 10 – 15% for majority of applications.

3. Conclusions

- Scale-up feasibility of nano-yttria dispersed CP Ti was successfully demonstrated by producing large quantities of modified alloys using conventional titanium powder metallurgy production processes.
- Addition of 0.3% Y_2O_3 increased the tensile strength of CP Ti by 25%.
- Addition of 0.5% B, in addition to 0.3% Y_2O_3, increased the tensile strength of CP Ti up to 67%.
- Addition of 0.5% B also increased the tensile modulus of CP Ti by 20% in addition to the significant strength increase while maintaining adequate tensile elongation. Yttria addition, on the other hand, provided only strength benefit to CP Ti.

Table 1: Room temperature tensile test data on yttria nano-particle reinforced CP Ti bars.

Alloy	ID	Test Log	Dia., mm	TYS		UTS		TE	TM	
				MPa	ksi	MPa	ksi	%	GPa	Msi
CP Ti	#2-1	2-1.txt	6.345	210	30.5	326.2	47.3	54.4	111.8	16.2
	#2-2	2-2.txt	6.317	198	28.7	323.9	47.0	47.8	103.2	15.0
	#2-3	2-3.txt	6.325	197.1	28.6	329.8	47.8	49.2	105.8	15.3
	#2-4	2.4.txt	6.322	193.3	28.0	331.6	48.1	47.4	106.8	15.5
	#2-5	2-5.txt	6.317	192.1	27.9	333.0	48.3	44.5	111.4	16.2
	#2-6	2-6.txt	6.314	184	26.7	334.3	48.5	53.1	97.5	14.1
			Avg	**196**	**28**	**329.8**	**48**	**49**	**106.1**	**15.4**
			Std Dev	8.6	1.2	4.0	0.6	3.7	5.4	0.8
CP Ti + 0.3 Y_2O_3	#1-1	1-1.txt	6.320	240.9	35	407.4	59.1	43.0	103.8	15.1
	#1-2	1-2.txt	6.322	245	36	409.7	59.4	43.5	105.7	15.3
	#1-3	1-3.txt	6.327	247.9	36	410.5	59.5	44.0	109.2	15.8
	#1-4	1-4.txt	6.335	248	36	409.7	59.4	49.6	103.8	15.1
	#1-5	1-5.txt	6.340	249	36	408.3	59.2	44.3	112.8	16.4
	#1-6	1-6.txt	6.325	251	36	412.9	59.9	46.8	106.6	15.5
			Avg	**247**	**36**	**410**	**59**	**45**	**107.0**	**15.5**
			Std Dev	3.6	0.5	1.9	0.3	2.5	3.5	0.5
CP Ti + 0.3 Y_2O_3 + 0.5 B	#3-1	3-1.txt	6.335	316	46	488.0	70.8	28.7	125.4	18.2
	#3-2	3-2.txt	6.320	332	48	495.7	71.9	27.0	128.5	18.6
	#3-3	3-3.txt	6.345	331	48	503.0	73.0	27.9	137.7	20.0
	#3-4	3-4.txt	6.335	328	48	505.2	73.3	28.0	114.5	16.6
	#3-5	3-5.txt	6.342	324	47	502.2	72.8	29.4	121.5	17.6
	#3-6	3-6.txt	6.330	328	48	503.1	73.0	27.3	134.5	19.5
			Avg	**327**	**47**	**500**	**72**	**28**	**127.0**	**18.4**
			Std Dev	5.9	0.8	6.5	0.9	0.9	8.5	1.2

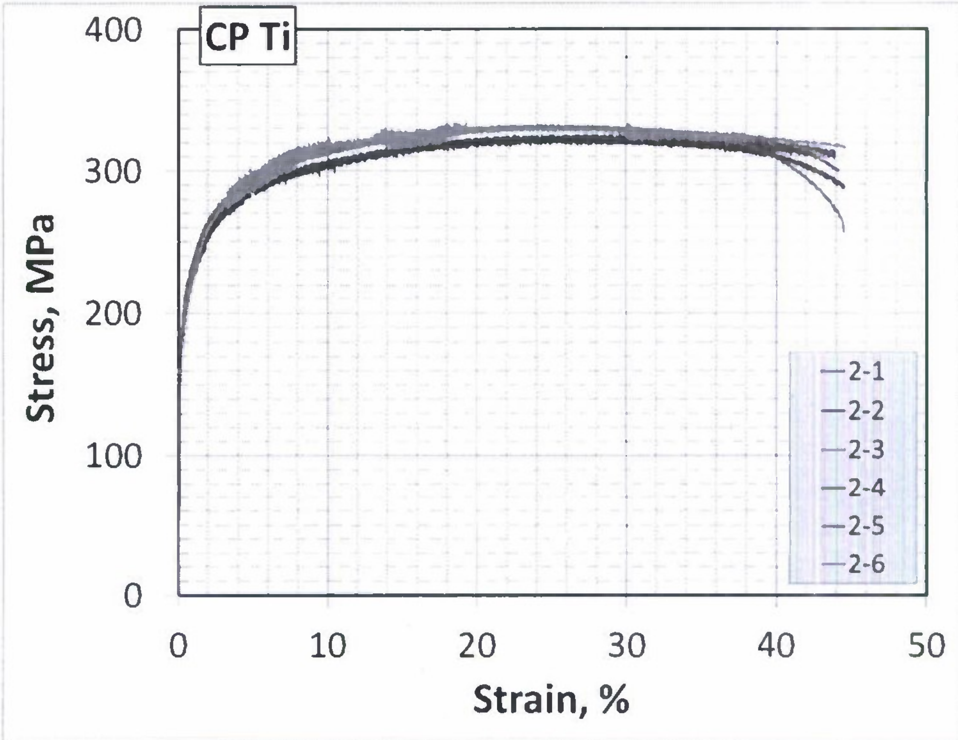

Figure 4: Tensile stress-strain curves at room temperature on CP Ti extruded bar.

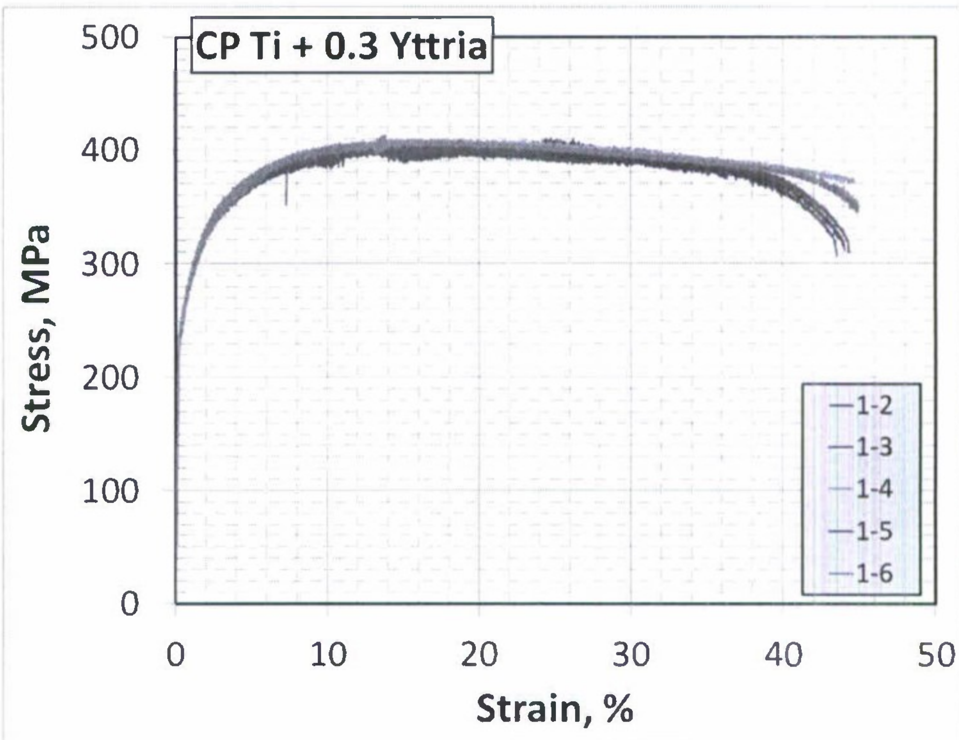

Figure 5: Tensile stress-strain curves at room temperature on CP Ti + 0.3 yttria extruded bar.

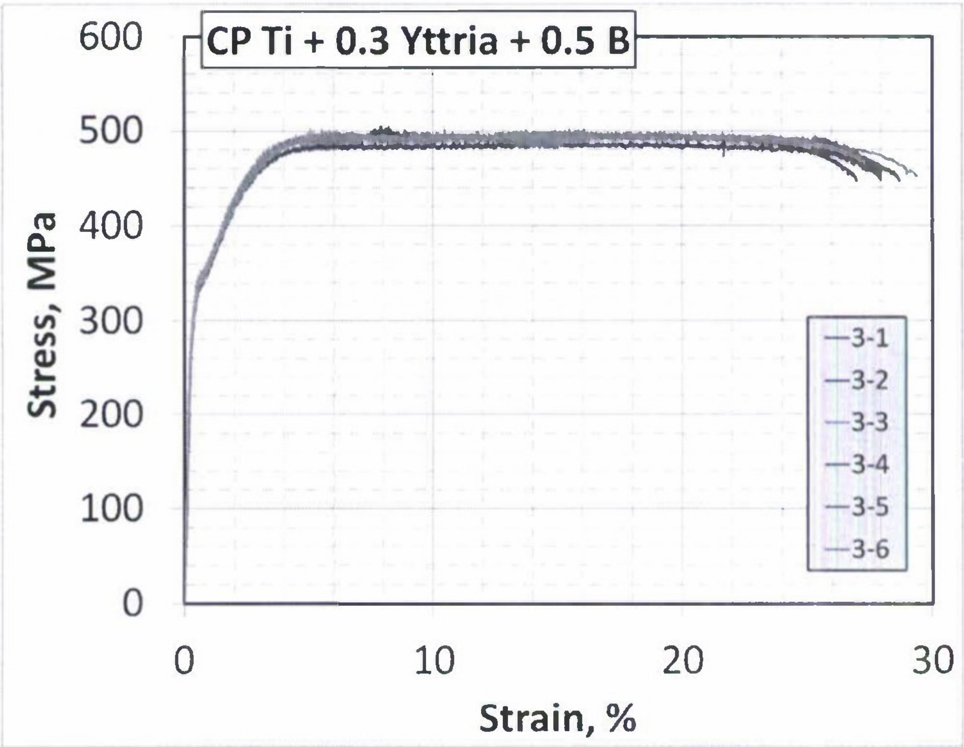

Figure 6: Tensile stress-strain curves at room temperature on CP Ti + 0.3 yttria + 0.5 B extruded bar.

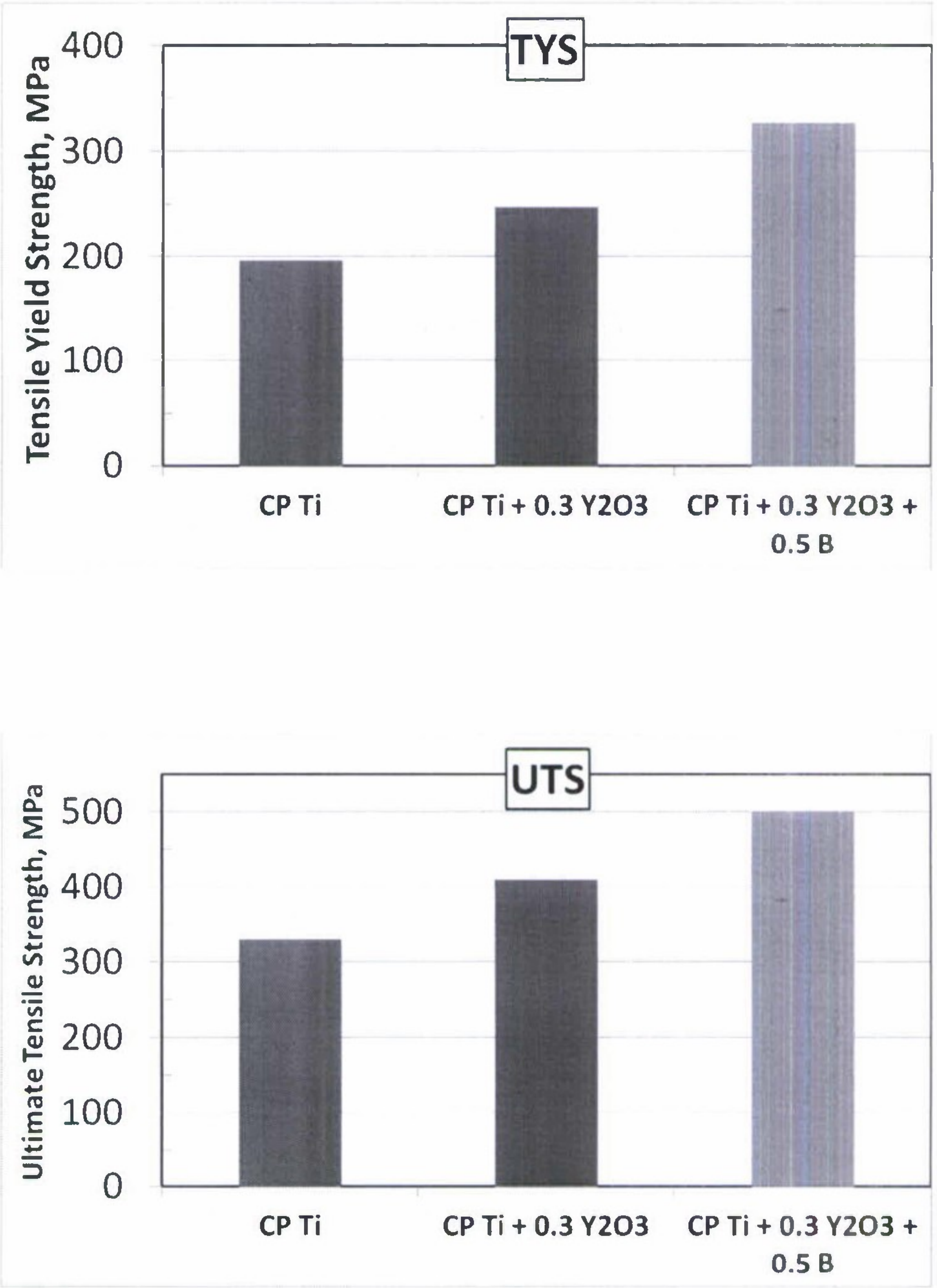

Figure 7: Comparison of tensile strengths (average values) of nano-yttria particle reinforced CP Ti extrusions.

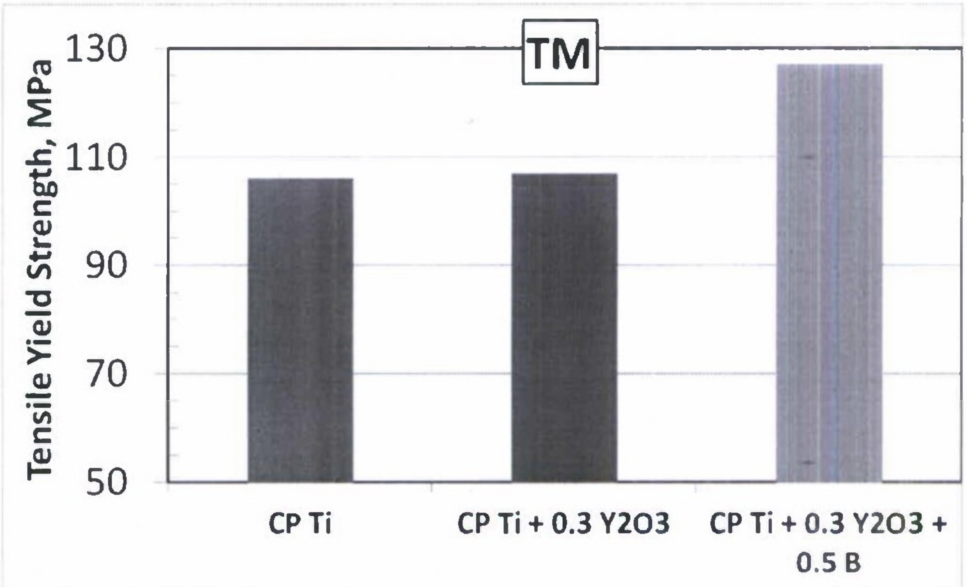

Figure 8: Comparison of tensile modulus (average values) of nano-yttria particle reinforced CP Ti extrusions.

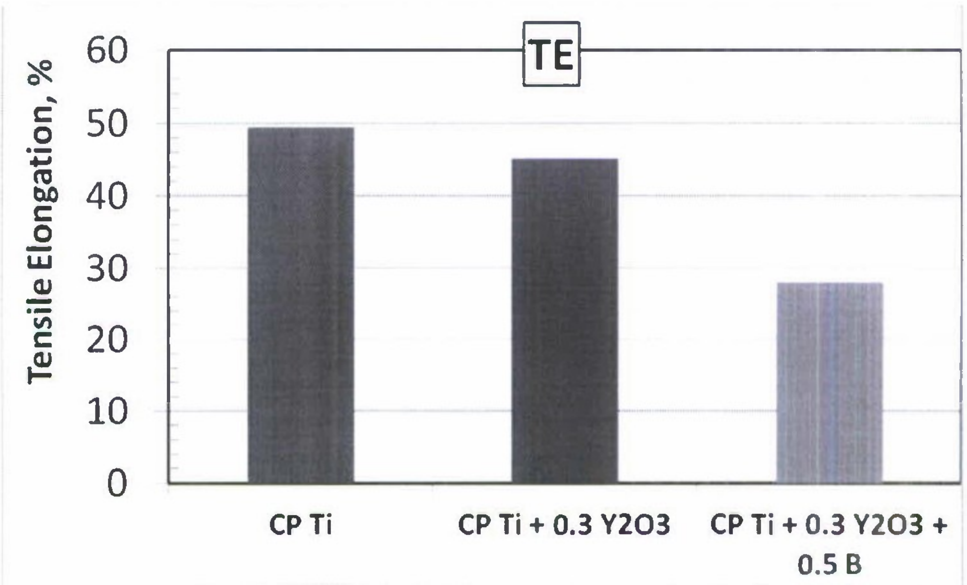

Figure 9: Comparison of tensile elongation (average values) of nano-yttria particle reinforced CP Ti extrusions.

4. Future Work

- Optimization of alloy chemistry to match the Fe and O contents to those in typical CP Ti Grade 2 could provide further improvements in tensile strength.
- Optimization of B content is necessary to obtain optimal property combinations in CP Ti.
- Microstructural evaluations are to be conducted to understand the structure-property relationships and to guide microstructural engineering.